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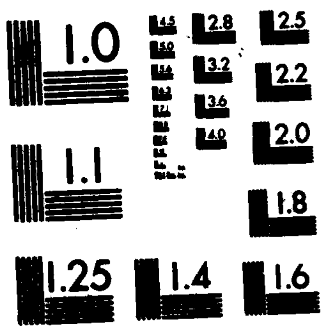

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

New multicomponent Zr free fluoride glasses are investigated for I.R. optical fiber preparation. A rotational casting technique associated with the fabrication of a new dry glove box have been developped in order to prepare tube and then core-clad preform. The glass BIZYT based on indium and barium essentially can be shaped as a tube without crystallization. Attempts for core-clad preform design indicate a tendency of crystallites formation at the core-clad interface. Contamination of the glass by transition metals and viscosity measurements are in a early stage of investigation. (Keywords)

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THIRD PERIODIC REPORT

In the first and second periodic reports, we described a new family of fluoride glasses called BIZYT, based on the multicomponent fluoride association $Ba_{20}In_{20}Zn_{20}Y_{10}Th_{10}$. The optical properties such as multiphonon edge in the 8 μm region, refractive index versus λ and composition, material dispersion, influence of OH^- impurities have been discussed in terms of potential applications of these glasses for I.R. optical fiber design. From this discussion, it was clear that the potential transparency of these glasses in the 4-5 μm region was about 10^2 better than the ZrF_4-based fibers which are limited by their multiphonon edge in this region.

In this third report, we will describe the premiminary results concerning : 1. The tentative of making preforms by rotational cations. 2. the influence of transition metal doping on the optical absorption and the problems related to viscosity.

1. Preforms preparation

First a large amount of time and energy have been used for the design of a cylindric mould which could be rotated at different speeds inside a glove box. It has been necessary to build a system allowing the transfert of the mould from vertical to horizontal positions during the rotation. This technique is largely inspired by the technology developped by SIEGEL and TRAN at N.R.L.

In the same time, a large dry glove box with about 1 m^3 volume in two compartments have been built essentially to treat and handle the molten fluoride in a very dry atmosphere. Previous corrosion studies have indicated that the corrosion of the melt by atmosphere moisture was very fast and critical for the contamination of the preforms by OH impurities.

In this new glove box, we are able to transfer the melt which is heated in a controlled atmosphere into the box without any contac· with

humidity. Two vitreous carbon crucibles containing for instance two different melts corresponding to the core and the clad can be introduced by this procedure into the box where the H_2O contamination is controlled and maintained in the range of few ppm.

In these first experiments, the fluoride glass melts have not been treated under any reactive atmosphere processing, but this operation will be possible in the future. Many experiments have been carried out in order to find the best conditions first to prepare a tube with a wall thickness of about 2 mm, and having about 10 cm length.

After the optimization of different parameters such as the temperature of the melt, the time of transfert in the box, the rotation speed of the mould, the position of the mould, it has been possible to prepare a BIZYT glass tube without apparent crystallization of the material. It appears that the critical parameters,as expected,is the viscosity of the melt. We do not have quantitative values of the viscosity versus temperature, but from the different observations, it is clear that the viscosity-temperature dependence is very severe and governs the quality of the experiments.

The best conditions for the moment have been found to be the following : the liquidus temperature of the BIZYT glass being 680° C, it is necessary to heat the melt first at 800° C, then keep the temperature at 750° C before transferring the melt in the box. During the short time (few minutes) between the transfert and the pouring in the mould, the melt temperature is such that no nucleation and crystallization seem to be apparent.

The rotational casting operation is followed by an annealing treatment of the mould containing the glass at 300° C in a furnace located inside the box.

Some other experiments related to the complete preparation of the preform have been also attempted. First, two crucibles containing two glass melts are transferred in the box. The tube is prepared following the previous procedure. Immediately after the rotation stops, the core glass is poured in the tube inserted in the mould. After solidification, the core-clad preform is annealed at 300° C.

At this step of the investigation, we can mentionned the following observations :

1. the preparation of a good quality tube is possible

2. the core-clad preform shows that the core is not crystallized but that formation of cristallites appears at the interface between the core and the clad

3. a simple tube could be prepared without apparent crystallization

4. all these preliminary experiments have been made with a mould of 13 mm inside diameter.

We just begin new investigations with a mould of 10 mm diameter in order to avoid the formation of cristallites at the core-clad interface.

2. Metallic impurities - viscosity.

In parallel with the preform fabrication, we also start the study of the effect of transition metal impurities on the optical absorption loss in the BIZYT. The following metals have been introduced in the glass with concentrations up to 2 % : V^{3+}, Cr^{3+}, Fe^{3+}, Ni^{2+}, Co^{2+}, Cu^{2+}, Fe^{2+}.

The absorption spectra have been recorded for all the doped glasses in the region 0.2 μm to 2.5 μm. Now, we have just begun the calculation of the ε value and the determination of the quantitative contaminating effect of these impurities for different wavelengths. This will be discussed in more details in the next report.

Also preliminary results obtained in collaboration with Pr. MOYNIHAN from R.P.I. (U.S.A.) and related to the viscosity measurements after Tg will be discussed in the next report.

At this time, we will be able to have a complete viscosity-temperature dependence curve from the solid to the liquid which will be extremely useful for determining the best conditions for preform preparation.

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